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## **PROVISIONAL SPECIFICATION**

**200390      Filed 21<sup>st</sup> November 2003**

**Invention Title : G I Tract Delivery Systems**

**Applicant: Australian Food Industry Science Centre and  
Commonwealth Scientific & Industrial Research  
Organisation**

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**The invention is described in the following statement:**

## G I Tract Delivery Systems

This invention relates to microencapsulated formulations for delivery of nutritional and pharmaceutical agents to the gastro intestinal tract and in particular the colon.

- 5 The compositions may be used for protection and delivery of nutrients or nutraceuticals in processed foods.

### Background to the invention

Microencapsulation involves the packaging of small particles of solid, liquid or gas 10 within a secondary material to form a microcapsule. It has been used for targeted delivery of drugs in the body in the pharmaceutical industry. It is increasingly being seen as a technology that offers novel food processing solutions. With the use of microencapsulation, possible undesirable interactions between the added nutraceutical and other components in the food or its environment can be avoided 15 and the site of release of the added component can be manipulated. The appropriate application of microencapsulation technology enables the fortification of food without affecting the taste, aroma or texture of food. It can afford protection to sensitive food ingredients and enhance the shelf-life and stability of fortified foods (Brazel, C.S. (1999) Microencapsulation: offering solutions for the food 20 industry. *Cereal Foods World* 44(6): 388-393; Augustin, M.A., Sanguansri, L., Margetts, C. and Young, B. (2001) Microencapsulation of food ingredients. *Food Australia* 53 220-223).

- Microencapsulation can serve both the purposes of the food and health industries, as it is a key technology with potential for the delivery of dietary bioactives and 25 development of successful marketable functional foods. Addressing this challenge, requires tailoring the performance of food grade microcapsules in a food processing environment so that essential sensitive components are protected during food manufacture and the microcapsules can also meet the need for site specific delivery within the gastrointestinal tract.
- 30 Directing nutraceuticals and therapeutics of the colon is of interest for treatment of colon diseases (Rubinstein, A., Tirosh, B., Baluom, M., Nassar, T., David, A., Radai, R., Gliko-Kabir, I. And Friedman, M. (1997). The rationale for peptide drug delivery to the colon and the potential for polymeric carriers as effective tools. *J.*

- Controlled Release 46, 59-73). Targeting to colon has been carried out by formation of pro-drugs which are enzymatically cleaved in the colon, and multi-coats with pH sensitive and pressure dependent release. Often enteric acrylic polymers are used to protect cores in colon-delivery formulations. Biopolymers, particularly polysaccharides, may be used for targeting cores to the colon where the release of cores is triggered by the microflora in the colon. A range of polysaccharides such as chitosan, pectin, arabinoxylan, arabinogalactan, xylan, cellulose dextrans, guar gum, amylose, inulin and mixtures of these have been examined and shown to have potential as colon-delivery systems (Rubinstein, A.
- 5 10 (2000) Natural Polysaccharides as targeting tools of drugs to the human colon. Drug Development Research 50, 435-439; Sinha, V.R. and Kumaria, R. (2001) Polysaccharides in colon-specific drug delivery *Int. J. Pharmaceutics* 224, 19-38; Vandaamme, Th.F., Lenourry, A., Charrueau, C. and Chaumeil, J.-C. (2002) The use of polysaccharides to target drugs to the colon. Carbohydrate Polymers 48, 15 219-231; Sinha, V.R. and Kumaria, R. (2003) Microbially triggered drug delivery to the colon. *Eur. J. Pharmaceutical Sciences* 18, 3-18).
- There has been a number of attempts to use biopolymers for colon delivery and for treating colonic diseases
- US Patent 5,952,314 discloses an enteral product comprising an oil blend with fatty acids {EPA (C20:5) and DHA(C22:6)} and a source of indigestible carbohydrate which is metabolised to short chain fatty acids in the colon. It has use for improving nutritional status and treating ulcerative colitis
- 20 US5108758 discloses a glassy amylose matrix for delivery of medication to the colon
- US 5840860 is concerned with delivery of short chain fatty acids (SCFA) to the colon by way of a modified starch.
- 25 Japanese patent 10324642 discloses a colon delivery system for delivery of bioactives (eg peptides) comprising inner layer of chitosan and outer-layer of gastric resistant material such as wheat gliadin or zein.
- US 5866619 discloses a colonic delivery system for drugs such as proteins and peptides comprising a saccharide containing polymer
- 30 US 6368629 discloses a drug coated with an organic acid-soluble polymer and a saccharide for colon delivery.

US 544054 discloses a method of treating colitis with a composition containing oil blend (with DHA/EPA) and a source of indigestible carbohydrate (CHO) which is metabolised to short chain fatty acids.

US 5952314 is concerned with an enteral nutritional product for treatment of colitis

- 5 which comprises oil containing EPA/DHA and a source of indigestible carbohydrate which is metabolised to short chain fatty acids.

US 6531152 describes a drug delivery system containing a water soluble core (Ca pectinate or other water-insoluble polymers) and outer coat which bursts (eg hydrophobic polymer - Eudragit) for delivery of enterally-administered drugs to

- 10 specific locations along the gastrointestinal tract

There are proposals using combinations of proteins and polysaccharides for the formation of coating systems.

US 6234464 discloses a system in which oils / polyunsaturated fatty acids (PUFA)/ fatty acids are provided with capsules comprised of two layers in which the inner

- 15 layer consists of gelatin, casein or alginate and the outer layer consists of gelatin, gum arabic, chitosan to provide a product stable in boiling water

US 6403130 discloses a coating composition comprising a polymer containing casein and high methoxy pectin (amide formed by reaction of ester group R'COOCH<sub>3</sub> of pectin with free amino group of protein R"NH<sub>2</sub>)

- 20 WO 01/74175 discloses the encapsulation of oxygen sensitive materials such as polyunsaturated oils in a protein carbohydrate film treated to form a Maillard reaction product.

It is an object of this invention to provide a gastrointestinal delivery system that can be used with storage unstable ingredients as well as providing protection during 25 delivery through the gut.

#### **Brief description of the Invention**

To this end the present invention provides a micro encapsulation material for use with storage unstable, therapeutic and nutritional agents which release the

- 30 therapeutic and nutritional agents in predetermined locations in the gastro intestinal tract in which the microencapsulation material is formed by combining a food grade treated carbohydrate with a water soluble food grade protein.

The therapeutic and nutritional agents form an oil phase which is emulsified with the water dispersed or dissolved encapsulant to encapsulate the therapeutic and nutritional agents. These agents may be oils or oil soluble or oil dispersible which in the latter case may include water soluble ingredients.

- 5 The agents that may be encapsulated include lipids (oils including oxygen sensitive oils, fatty acids, triglycerides) and oil soluble and oil dispersible ingredients (including pharmaceuticals, probiotics, protein therapeutics and bioactives). Water soluble or water dispersible components including those that partition between oil and water phases may also be encapsulated. When water
  - 10 soluble or water dispersible therapeutic and nutritional agents are used they may not be encapsulated with the oil phase but may be dispersed in the encapsulant film. The emulsions may be used as food ingredients or therapeutic agents but preferably the emulsions are dried to form powders.
- Prior art encapsulation systems did not consider the use of combinations of
- 15 proteins with other biopolymers for formation of capsules for target delivery of sensitive cores to the colon.

- The delivery systems of this invention enable pharmaceutical and food manufacturers to offer a range of nutritionally and physiologically functional food ingredients and bioactive compounds in convenient formats and using all natural ingredients which will also enable the delivery of these products to the colon.
- 20 Some of the encapsulants used for colon delivery in this invention have the added benefits of being effective matrices for encapsulating oxygen sensitive ingredients. The film-forming and anti-oxidant properties of some of the encapsulants used work synergistically to preserve sensitive ingredients such as polyunsaturated fatty acids from being oxidised during storage and also protects them during exposure to high temperature, pressure and moisture encountered during the processing of foods. In addition, this invention uses readily available proteins and carbohydrates.
- 25 There are no solvents used in the preparation of the encapsulated formulations as the process is an all-aqueous based system. The processes can be easily incorporated or adapted to suit most food and pharmaceutical manufacturing plants with drying operations.
- 30 The protein used may include any film forming water soluble protein or hydrolysed protein and includes milk proteins such as casein and its derivatives or whey

proteins. The carbohydrate component may be those containing reducing sugar groups, oligosaccharides and starches (raw, modified, resistant, acetylated, propionate and butyrate starches).

- The proteins and carbohydrates may be reacted in aqueous solutions to obtain
- 5 conjugates. The reaction, which occurs, can be between free amine groups of amino acids in the protein and reducing sugar groups in the carbohydrate. This type of reaction is generally termed a Maillard reaction typically occurring in the non-enzymatic browning of foods. This reaction occurs during heat processing of foods and has previously been shown to be beneficial for engineering desirable
- 10 encapsulating properties for protection of oxygen sensitive components. For example, microencapsulated formulations containing oxygen sensitive oils are protected against oxidation as the Maillard reaction products [MRP] in the encapsulating matrix are good film-formers and also exhibit anti-oxidation activity as disclosed in WO 01/74175 .
- 15 The starches used in the formulations may also be pre-processed using conventional and emerging processing technologies to modify the starch properties to provide improved processing characteristics during the preparation of the delivery systems. The pretreatments are chosen to break down the long starch molecules so that they form more stable emulsions and also to provide a larger
- 20 number of terminal sugar reducing groups for Maillard reaction with the protein component of the encapsulant.
- Colon delivery systems may be used for range of bioactives (eg oils), pharmaceuticals and therapeutics (eg proteins, peptides and vaccines), which are unstable in the upper gastrointestinal tract. The protection afforded to the
- 25 encapsulated components by the encapsulating material enable target release in the colon where the release is achieved after the encapsulant is degraded (eg by the action of microbial enzymes in the colon). Delivery of bioactives, pharmaceuticals and therapeutic components to the colon is desirable for treatment and prevention of diseases of the colon such as colorectal cancer,
- 30 ulcerative colitis and inflammatory bowel disorder.
- In some cases the encapsulants used in the formulations such as selected polysaccharides, can also serve as gut wall adherens or as prebiotics that facilitate growth of beneficial bacteria, and can offer added advantages. For example

delivery systems containing resistant starch have potential benefits on colonic health.

#### Detailed Description of the invention

- 5 A number of formulations will be described, some according to the invention and some for comparative purposes to show that some formulations are suitable to delivery to the colon whilst others could be more suitable for release in the small intestine. These formulations demonstrate that the core is protected from digestion in the stomach and the environment in the small intestine.
- 10 Figures 1 to 15 of the drawings graphically illustrate the solvent extractable fat content and other properties of the formulations of the invention as illustrated in examples 1 to 15 below.

The process of microencapsulating the active component involves the following manufacturing steps:

- 15 (a) Selection of the biologically active core (eg oil, oil soluble or oil dispersible material, bioactives, therapeutics, pharmaceuticals)
- (b) Dispersion of the protein and carbohydrates (or starch that has been pre-processed by conventional means such as heating or extrusion or by the use of emerging processing technologies such as high pressure processing, microfluidisation or ultrasonics) in the aqueous phase and treatment of the mixture. If desired, the protein-carbohydrate blends may be further heat processed to induce the formation of conjugates (eg Maillard reaction products)
- 20 (c) Mixing the core with the encapsulant (ie protein-carbohydrate mixture) and homogenizing the mixture to obtain an emulsion
- (d) Spray drying the emulsion to obtain a powdered formulation in which the core is surrounded by the encapsulating matrix

#### Emulsion formulations

- 30 Tuna fish oil was used as an oil of choice in most of these examples since it contains a high amount of long chain polyunsaturated fatty acids and this need to be protected from oxidation prior to consumption. In addition there is interest in delivering these to the colon because of their potential for prevention of colorectal

- cancer and promotion of bowel health (Karmeli, R A. (1996) Historical Perspective and Potential Use of n-3 Fatty Acids in Therapy of Cancer Cachexia. *Nutrition*, Vol 12 (1) S2-S4; Dommels Y E M, Alink, G M, van Bladeren, P J, van Ommen, B (2002) Dietary n-6 and n-3 polyunsaturated fatty acids and colorectal
- 5 carcinogenesis: results from cultured colon cells, animal models and human studies, *Environmental Toxicology and Pharmacology*, Vol 12 (4), 233-244.) A range of formulations was prepared using protein and/or carbohydrate (raw or pre-processed) and oil mixtures at different ratios. The formulations were made-up to contain 25 and 50% fat in the final powder.
- 10 The protein used in these examples was sodium caseinate, whey protein isolate and hydrolysed milk proteins. The carbohydrates used, alone or in combination, were glucose, oligosaccharides, dried glucose syrup, modified starches, resistant starches). Polysaccharides, including high-methoxy pectin, alginate, carrageenan, guar gum, were added to protein-carbohydrate mixtures in some formulations.

15

**Examples: Manufacture of microcapsules****Materials for preparation of microcapsules**

- Tuna oil (HIDHA® 25N FOOD – steam deodorised) was obtained from Clover Corporation Ltd. Sodium Caseinate (Alanate 180) (NaCas) and whey protein
- 20 isolate (Alacen 895) (WPI) were from New Zealand Milk Products, NZ. Hydrolysed casein protein (HCP 102) and hydrolysed whey protein (HWP 205) were from Tatua Co-Operative Dairy Company, Ltd, NZ.
- Dextrose monohydrate (Glu), waxy maize and maize starch were from Penford Australia, Au. Dried glucose syrup (Maltostar 30) (DGS) and wheat starch were
- 25 from Manildra, Au. Oligofructose (Raftilose P95) (oligo) was from Mandurah Australia Pty Ltd. Tapioca dextrin (K4484), modified starch (Capsul), modified starch (Hi-Cap 100), Hi-Maize, Hylon VII, Novelose 260 and Novelose 330 was from National Starch and Chemical Co. Potato starch, sodium alginate (Protanal SF120RB) and kappa carrageenan (Gelcarin GP812) was from Swift and Co Ltd.
- 30 High methoxy pectin (RS 400) (HMP) was from Danisco and guar gum (WW250F) from Woods & Woods Pty Ltd.

#### Preparation of protein-carbohydrate encapsulants

In some cases, unreacted blends of protein and carbohydrates (referred to as NonMRP formulations since these were not heated to induce the formation of Maillard reaction products) were used as the encapsulating matrix for the preparation of reacted protein-carbohydrate encapsulants (referred to as MRP formulations as these were heated to induce the formation of Maillard reaction products), protein was dissolved in 60°C water, using a high shear mixer, and then the sugars, starch or the selected carbohydrate were added. Where a polysaccharide was also added, the polysaccharide was first allowed to hydrate in water at 90°C temperature before addition into the protein-sugar mixture. The pH of the protein-sugar/starch /gum mixtures was adjusted to 7.5. The mixture were then filled into 3 litre cans, sealed and heated in the retort to 98°C and held for 30 minutes, then cooled down to room temperature. Microcapsule formulations are given in the examples below together with the methods used for the manufacture of microcapsules.

#### Preparation of protein-starch encapsulants

Protein was dissolved in 60°C water to make 15% total solids (TS) solution, using a high shear mixer. Starch (raw or heated, heat and microfluidised, extruded, high pressure processed and ultrasonicated) was prepared and processed separately to make a 10% TS solutions or dispersions in 70°C water (See Preparation of Starches for Microencapsulation detailed below). The 15%TS protein solution were mixed together with the 10%TS starch to get a 12%TS mixture with a 1:1 protein:starch ratio. Where MRP was required, the mixture were then filled into 3 litre cans, sealed and heated in the retort to 98°C and held for 30 minutes, then cooled down to 60°C.

#### **Preparation of Starches for Microencapsulation**

##### Raw

10% TS starch dispersion (no pre-treatment applied) was mixed with 15% TS of protein solution at 60°C.

##### 30 Heat processing

20% TS of each starch dispersion (except for potato starch where a 10% TS dispersion was used due to high viscosity at 20% TS) were heated at 121°C for 60 minutes in a 73x82 mm cans. Once heat processed, 70°C deionised water was

added to dilute the sample to 10% TS in a high shear mixer. This heat processed starch was mixed with 15% TS of protein solution at 60°C. This mixture was then used for microencapsulation of bioactives.

Heat processing and Microfluidisation treatment

- 5    20% TS of each starch dispersion (except for potato starch where a 10% TS dispersion was used due to high viscosity at 20% TS) were heated at 121°C for 60 minutes in a 73x82 mm cans. Once heat processed, 70°C deionised water was added to dilute the sample to 10% TS in a high shear mixer, and processed at 60°C through a pilot scale M-210B EH microfluidiser (MFIC, Newton MA, USA).
- 10   The plant was operated at 800 bars and 3 passes using a combination of 425 µm Q50Z auxiliary processing module and 200 µm E230Z interaction chamber (for dispersion and cell disruption). The microfluidised (MF) starch was mixed with 15% TS of protein solution at 60°C for microencapsulation.

Heat processing and Ultra-high pressure treatment

- 15   20% TS of a starch dispersion was heated at 121°C for 60 minutes in a 73x82 mm cans. Once heat processed, 70°C deionised water was added to dilute the sample to 10% TS in a high shear mixer, and processed by ultra-high pressure treatment at 6,000 bars for 15 minutes using HPP-QFP 35L unit. The ultra-high pressure treated (HPP) starch was mixed with 15% TS of protein solution at 60°C for microencapsulation.

Heat processing and Ultrasonics treatment

- 20   20% TS of a starch dispersion was heated at 121°C for 60 minutes in a 73x82 mm cans. Once heat processed, 70°C deionised water was added to dilute the sample to 10% in a high shear mixer, and processed with ultrasound treatment at 25   50 ml/min @ 380 watts using 20KHz unit. The ultrasound treated (US) starch was mixed with 15% TS of protein solution at 60°C for microencapsulation.

Extrusion

- Resistant starches were processed using a twin-screw extruder (model MPF 40, APV Baker, Peterborough PE3-6TA, England) 40mm screw diameter and length to diameter ratio of 25:1, and a low shear screw configuration. A 4 mm die was used throughout the trial. Raw materials were fed into feed port 1 at 15 kg h<sup>-1</sup> for resistant starch processing using a gravimetric feeder (Ktron Soder AG CH-5702, Niederlenz) and water was injected into port 2 with a volumetric pump (Brook Crompton, Huddersfield, England). Barrel

moisture was injected at 20-40 % and the die melt temperature was varied from 140 to 178°C with increasing screw speed from 150-250rpm. The extruded resistant starches were milled to 0.2mm particle size powder. 10% TS extruded starch dispersion was mixed with 15% TS of protein solution at 60°C for microencapsulation.

5 **Preparation of tuna oil emulsions**

The protein-carbohydrate mixtures and the tuna oil were pre-heated to 60°C separately. The tuna oil was added into the protein-carbohydrate mixture using a Silverson high shear mixer. The mixture were then homogenised at 350 and 100 bar pressures in two stages using a Rannie homogeniser.

10 **Spray drying of emulsions**

The homogenised emulsions were spray dried at 50-60°C feed temperature, 180°C inlet temperature and 80°C outlet temperature using a Niro production minor spray dryer. The powder was collected from the main chamber and packed.

15 **Estimation of solvent extractable fat in powders**

The estimation of solvent-extractable was based on the method by Pisecky (Handbook of Milk Powder Manufacture, 1997) except that petroleum ether was used in place of carbon tetrachloride. Fifty ml of petroleum ether (b.p. 40-60°C) was added to 10g powder. The mixture was agitated in a stoppered flask for 15 minutes. The mixture was filtered and the solvent evaporated at 60°C using a rotary evaporator. The remaining fat residue was then dried in an oven at 105°C for 1h.

**In-vitro testing of microcapsules**

The stability of the microcapsules in the stomach and the small-intestine was estimated by assessment of oil-release properties of microcapsules (a) incubated in simulated gastric fluid (SGF) (pH 1.2) for 2h at 37°C and 100 rpm in a shaker water-bath incubator and (b) incubated in SGF (2 h at 37°C and 100 rpm in a shaker water-bath incubator) followed by exposure to simulated intestinal fluid (SIF) (pH 6.8) (3 h at 37°C and 100 rpm). SGF and SIF were prepared according to the methods given in the US Pharmacopia (US Pharmacopia 2000 & National Formulatory (USP 24 NF 19), Rockville, MD)

For estimation of released oil from microcapsules, the solvent extractable fat from the incubated samples were measured. The sample was transferred into a 250 ml stoppered separating funnel and extracted with petroleum ether (75 ml plus 2x25ml). The sample was filtered through a phase separation filter paper to obtain the solvent phase after each extraction. The solvent was removed to recover the oil released.

## Determination of Resistant Starch Content

The fat content of resistant starch was measured using the Megazyme Resistant Starch Assay Procedure (RSTAR 11/02 AOAC Method 2002-02; AACC Method 54-10).

10 32-40).

**Example 1: Formulations and Manufacture of Powders with 25% oil loading with unheated or heated blends of protein- glucose/dried glucose syrup or protein- oligosaccharide as encapsulants**

15

NaCas-Glucose-DGS-NonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		69.2%	4500	Prepare NaCas solution at 60°C, add sugars, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Inlet temperature (Ti)/Outlet temperature (To).
Alanate 180	25.0%	7.7%	500	
Glucose.H <sub>2</sub> O	25.0%	7.7%	500	
Maltostar 30	25.0%	7.7%	500	
Tuna oil	25.0%	7.7%	500	
Total	100.0%	100.0%	6500	

NaCas-Glucose-DGS-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		69.2%	4500	Prepare NaCas solution at 60°C, add sugars, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Alanate 180	25.0%	7.7%	500	
Glucose.H <sub>2</sub> O	25.0%	7.7%	500	
Maltostar 30	25.0%	7.7%	500	
Tuna oil	25.0%	7.7%	500	
Total	100.0%	100.0%	6500	

NaCas-oligosaccharide-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		69.2%	4500	Prepare NaCas solution at 60°C, add oligosaccharide, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C
Alanate 180	25.0%	7.7%	500	Ti/To.
Raftilose P95	50.0%	15.4%	1000	
Tuna oil	25.0%	7.7%	500	
Total	100.0%	100.0%	6500	

WPI-glucose-DGS-NonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		69.2%	4500	Prepare WPI solution at 60°C, add sugars, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Alacen 895	25.0%	7.7%	500	
Glucose.H <sub>2</sub> O	25.0%	7.7%	500	
Maltostar 30	25.0%	7.7%	500	
Tuna oil	25.0%	7.7%	500	
Total	100.0%	100.0%	6500	

WPI-glucose-DGS-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		69.2%	4500	Prepare WPI solution at 60°C, add sugars, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Alacen 895	25.0%	7.7%	500	
Glucose.H <sub>2</sub> O	25.0%	7.7%	500	
Maltostar 30	25.0%	7.7%	500	
Tuna oil	25.0%	7.7%	500	
Total	100.0%	100.0%	6500	

WPI-oligosaccharide-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		69.2%	4500	Prepare WPI solution at 60°C, add sugars, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Alacen 895	25.0%	7.7%	500	
Raftilose P95	50.0%	15.4%	1000	
Tuna oil	25.0%	7.7%	500	
Total	100.0%	100.0%	6500	

- Example 2: Formulations and Manufacture of Powders with 50% oil loading with unheated or heated blends of protein- glucose/dried glucose syrup or protein- oligosaccharide as encapsulants**

NaCas-glucose-DGS-NonMRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.0%	4500	Prepare NaCas solution at 60°C, add sugars, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Alanate 180	16.7%	6.7%	500	
Glucose.H <sub>2</sub> O	16.7%	6.7%	500	
Maltostar 30	16.7%	6.7%	500	
Tuna oil	50.0%	20.0%	1500	
Total	100.0%	100.0%	7500	

NaCas-glucose-DGS-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.0%	4500	Prepare NaCas solution at 60°C, add sugars, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Alanate 180	16.7%	6.7%	500	
Glucose.H <sub>2</sub> O	16.7%	6.7%	500	
Maltostar 30	16.7%	6.7%	500	
Tuna oil	50.0%	20.0%	1500	
Total	100.0%	100.0%	7500	

NaCas-oligosaccharide-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.0%	4500	
Alanate 180	16.7%	6.7%	500	
Raftilose P95	33.3%	13.3%	1000	
Tuna oil	50.0%	20.0%	1500	
Total	100.0%	100.0%	7500	Prepare NaCas solution at 60°C, add oligosaccharide, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

WPI-glucose-DGS-NonMRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.0%	4500	
Alacen 895	16.7%	6.7%	500	
Glucose.H <sub>2</sub> O	16.7%	6.7%	500	
Maltostar 30	16.7%	6.7%	500	
Tuna oil	50.0%	20.0%	1500	
Total	100.0%	100.0%	7500	Prepare WPI solution at 60°C, add sugars, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

WPI-glucose-DGS-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.0%	4500	
Alacen 895	16.7%	6.7%	500	
Glucose.H <sub>2</sub> O	16.7%	6.7%	500	
Maltostar 30	16.7%	6.7%	500	
Tuna oil	50.0%	20.0%	1500	
Total	100.0%	100.0%	7500	Prepare WPI solution at 60°C, add sugars, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

WPI-oligosaccharide-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.0%	4500	
Alacen 895	16.7.0%	6.7%	500	
Raftilose P95	33.3%	13.3%	1000	
Tuna oil	50.0%	20.0%	1500	
Total	100.0%	100.0%	7500	Prepare WPI solution at 60°C, add sugars, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

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**Example 3: Formulations and Manufacture of Powders with 25% oil loading with heated blends of protein- starch as encapsulants**

NaCas-Capsul-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		69.2%	4500	
Alanate 180	25.0%	7.7%	500	
Capsul	50.0%	15.4%	1000	
Tuna oil	25.0%	7.7%	500	
Total	100.0%	100.0%	6500	Prepare NaCas solution at 60°C, add starch, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-HiCap-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		69.2%	4500	
Alanate 180	25.0%	7.7%	500	
Hi-Cap 100	50.0%	15.4%	1000	
Tuna oil	25.0%	7.7%	500	
Total	100.0%	100.0%	6500	Prepare NaCas solution at 60°C, add starch, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-K4484-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		69.2%	4500	
Alanate 180	25.0%	7.7%	500	
Tapioca dextrin K4484	50.0%	15.4%	1000	
Tuna oil	25.0%	7.7%	500	
Total	100.0%	100.0%	6500	Prepare NaCas solution at 60°C, add dextrin, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

WPI-K4484-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		69.2%	4500	
Alacen 895	25.0%	7.7%	500	
Tapioca dextrin K4484	50.0%	15.4%	1000	
Tuna oil	25.0%	7.7%	500	
Total	100.0%	100.0%	6500	Prepare WPI solution at 60°C, add dextrin, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

5 **Example 4: Formulations and Manufacture of Powders with 50% oil loading with heated blends of protein- starch as encapsulants**

NaCas-Capsul-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.0%	4500	
Alanate 180	16.7%	6.7%	500	
Capsul	33.3%	13.3%	1000	
Tuna oil	50.0%	20.0%	1500	
Total	100.0%	100.0%	7500	Prepare NaCas solution at 60°C, add starch, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-HiCap-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.0%	4500	
Alanate 180	16.7%	6.7%	500	
Hi-Cap 100	33.3%	13.3%	1000	
Tuna oil	50.0%	20.0%	1500	
Total	100.0%	100.0%	7500	Prepare NaCas solution at 60°C, add starch, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-K4484-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.0%	4500	
Alanate 180	16.7%	6.7%	500	
Tapioca dextrin K4484	33.3%	13.3%	1000	
Tuna oil	50.0%	20.0%	1500	
Total	100.0%	100.0%	7500	

WPI-K4484-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.0%	4500	
Alacen 895	16.7%	6.7%	500	
Tapioca dextrin K4484	33.3%	13.3%	1000	
Tuna oil	50.0%	20.0%	1500	
Total	100.0%	100.0%	7500	

**Example 5: Formulations and Manufacture of Powders with 25% oil loading with heated blends of protein-glucose/glucose syrup or protein–oligosaccharide in combination with gums as encapsulants**

NaCas-glu-DGS-alginate-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		69.2%	4500	
Alanate 180	25.0%	7.7%	500	
Glucose.H <sub>2</sub> O	25.0%	7.7%	500	
Maltostar 30	22.5%	6.9%	450.0	
Protanal	2.5%	0.8%	50.0	
Tuna oil	25.0%	20.0%	500	
Total	100.0%	100.0%	6500	

NaCas-oligosaccharide-guar-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		50.1%	4500	
Alanate 180	25.0%	5.6%	500	
Raftilose P95	48.75%	10.9%	975	
Guar WW250F	1.25%	0.3%	25	
Water for gum dispersion		27.6%	2475	
Tuna oil	25.0%	5.6%	500	
Total	100.0%	100.0%	8975	

NaCas-oligosaccharide-carrageenan-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.2%	4500	Prepare NaCas solution at 60°C, add oligosaccharide and carrageenan solution, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Alanate 180	25.0%	6.7%	500	
Raftilose P95	48.75%	13.0%	975	
Gelcarin GP 812	1.25%	0.3%	25	
Water for gum dispersion		13.0%	975	
Tuna oil	25.0%	6.7%	500	
Total	100.0%	100.0%	7475	

NaCas-oligosaccharide-HMP-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.4%	4500	Prepare NaCas solution at 60°C, add oligosaccharide and high methoxy pectin (HMP) solution, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
NaCas	25.0%	6.7%	500	
Raftilose P95	47.5%	12.7%	950	
HMP RS400	2.5%	0.7%	50.0	
Water for gum dispersion		12.7%	950	
Tuna oil	25.0%	6.7%	500	
Total	100.0%	100.0%	7450	

WPI-oligosaccharide-guar-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		50.1%	4500	Prepare WPI solution at 60°C, add oligosaccharide, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add 60°C guar gum solution, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Alacen 895	25.0%	5.6%	500	
P95	48.75%	10.9%	975	
Guar WW250F	1.25%	0.3%	25	
Water for gum dispersion		27.6%	2475	
Tuna oil	25.0%	5.6%	500	
Total	100.0%	100.0%	8975	

WPI-oligosaccharide-carrageenan-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.2%	4500	Prepare NaCas solution at 60°C, add oligosaccharide, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add 60°C carrageenan solution, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Alacen 895	25.0%	6.7%	500	
Raftilose P95	48.75%	13.0%	975	
Gelcarin GP 812	1.25%	0.3%	25	
Water for gum dispersion		13.0%	975	
Tuna oil	25.0%	6.7%	500	
Total	100.0%	100.0%	7475	

WPI-oligosaccharide-HMP-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
water		60.4%	4500	
Alacen 895	25.0%	6.7%	500	
Raftilose P95	47.5%	12.7%	950	
HMP RS400	2.5%	0.7%	50	
Water for gum dispersion		12.7%	950	
Tuna oil	25.0%	6.7%	500	
Total	100.0%	100.0%	7450	Prepare NaCas solution at 60°C, add oligosaccharide, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add 60°C HMP solution, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

**Example 6: Formulations and Manufacture of Powders with 50% oil loading with heated blends of protein-glucose/glucose syrup or protein-oligosaccharide in combination with gums as encapsulants**

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NaCas-Glucose-DGS-alginate-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
water		60.0%	4500	
Alanate 180	16.7%	6.7%	500	
Glucose	16.7%	6.7%	500	
Maltostar 30	15.0%	6.0%	450	
Protanal	1.7%	0.7%	50	
Tuna oil	50.0%	20.0%	1500	
Total	100.0%	100.0%	7500	Prepare NaCas solution at 60°C, add sugars and alginate (Protanal), adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-oligosaccharide-guar gum-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		45.1%	4500	
NaCas	16.7%	5.0%	500	
Raftilose P95	32.5%	9.8%	975	
Guar WW250F	0.8%	0.3%	25	
Water for gum dispersion		24.8%	2475	
Tuna oil	50.0%	15.0%	1500	
Total	100.0%	100.0%	9975	Prepare HWP solution at 60°C, add oligosaccharide and guar gum solution, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-oligosaccharide-carrageenan-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		53.1%	4500	
Alanate 180	16.7%	5.9%	500	
Raftilose P95	32.5%	11.5%	975	
Gelcarin GP 812	0.8%	0.3%	25	
Water for gum dispersion		11.5%	975	
Tuna oil	50.0%	17.7%	1500	
Total	100.0%	100.0%	8475	Prepare NaCas solution at 60°C, add oligosaccharide and carrageenan solution, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-oligosaccharide-HMP-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		53.3%	4500	
Alanate 180	16.7%	5.9%	500	
Raftilose P95	31.7%	11.2%	950	
HMP RS400	1.7%	0.6%	50	
Water for gum dispersion		11.2%	950	
Tuna oil	50.0%	17.8%	1500	
Total	100.0%	100.0%	8450	

WPI-oligosaccharide-guar gum-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		45.1%	4500	
Alacen 895	16.7%	5.0%	500	
P95	32.5%	9.8%	975	
Guar WW250F	0.8%	0.3%	25	
Water for gum dispersion		24.8%	2475	
Tuna oil	50.0%	15.0%	1500	
Total	100.0%	100.0%	9975	

WPI-oligosaccharide-carrageenan-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		53.1%	4500	
Alacen 895	16.7%	5.9%	500	
Raftilose P95	32.5%	11.5%	975	
Gelcarin GP 812	0.8%	0.3%	25	
Water for gum dispersion		11.5%	975	
Tuna oil	50.0%	17.7%	1500	
Total	100.0%	100.0%	8475	

WPI-oligosaccharide-HMP-MRP (50% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		53.3%	4500	
Alacen 895	16.7%	5.9%	500	
Raftilose P95	31.7%	11.2%	950	
HMP RS400	1.7%	0.6%	50.0	
Water for gum dispersion		11.2%	950	
Tuna oil	50.0%	17.8%	1500	
Total	100.0%	100.0%	8450	

**Example 7: Formulations and Manufacture of Powders with 25% oil loading with heated blends of protein hydrolysate - oligosaccharide in combination with gums as encapsulants**

<b>HCP-oligosaccharide-carrageenan-MRP (25% tuna oil in powder)</b>				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.2%	4500	
HCP 102	25.0%	6.7%	500	
Raftilose P95	48.75%	13.0%	975	
Gelcarin GP 812	1.25%	0.3%	25	
Water for gum dispersion		13.0%	975	
Tuna oil	25.0%	6.7%	500	
Total	100.0%	100.0%	7475	Prepare hydrolysed casein protein (HCP) solution at 60°C, add oligosaccharide, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add 60°C carrageenan solution, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

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<b>HCP-oligosaccharide-HMP-MRP (25% tuna oil in powder)</b>				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.4%	4500	
HCP 102	25.0%	6.7%	500	
P95	47.5%	12.7%	950	
HMP RS400	2.5%	0.7%	50	
Water for gum dispersion		12.7%	950	
Tuna oil	25.0%	6.7%	500	
Total	100.0%	100.0%	7450	Prepare HCP solution at 60°C, add oligosaccharide and HMP solution, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

<b>HWP-oligosaccharide-carrageenan-MRP (25% tuna oil in powder)</b>				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.2%	4500	
HWP 205	25.0%	6.7%	500	
Raftilose P95	48.75%	13.0%	975	
Gelcarin GP 812	1.25%	0.3%	25	
Water for gum dispersion		13.0%	975	
Tuna oil	25.0%	6.7%	500	
Total	100.0%	100.0%	7475	Prepare hydrolysed whey protein (HWP) solution at 60°C, add oligosaccharide and carrageenan solution, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

<b>HWP-oligosaccharide-HMP-MRP (25% tuna oil in powder)</b>				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		60.4%	4500	
HWP 205	25.0%	6.7%	500	
Raftilose P95	47.5%	12.7%	950	
HMP RS400	2.5%	0.7%	50	
Water for gum dispersion		12.7%	950	
Tuna oil	25.0%	6.7%	500	
Total	100.0%	100.0%	7450	Prepare HWP solution at 60°C, add oligosaccharide and HMP solution, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

**Example 8: Formulations and Manufacture of Powders with 50% oil loading with heated blends of hydrolysate - oligosaccharide in combination with gums as encapsulants**

<b>HCP-oligosaccharide-carrageenan-MRP (50% tuna oil in powder)</b>				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		53.1%	4500	
HCP 102	16.7%	5.9%	500	
Raftilose P95	32.5%	11.5%	975	
Gelcarin GP 812	0.8%	0.3%	25	
Water for gum dispersion		11.5%	975	
Tuna oil	50.0%	17.7%	1500	
Total	100.0%	100.0%	8475	Prepare HCP solution at 60°C, add oligosaccharide and carrageenan solution, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

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<b>HCP-oligosaccharide-HMP-MRP (50% tuna oil in powder)</b>				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		53.3%	4500	
HCP 102	16.7%	5.9%	500	
Raftilose P95	31.7%	11.2%	950	
HMP RS400	1.7%	0.6%	50.0	
Water for gum dispersion		11.2%	950	
Tuna oil	50.0%	17.8%	1500	
Total	100.0%	100.0%	8450	Prepare HCP solution at 60°C, add oligosaccharide and HMP solution, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

<b>HWP-oligosaccharide-carrageenan-MRP (50% tuna oil in powder)</b>				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		53.1%	4500	
HWP 205	16.7%	5.9%	500	
Raftilose P95	32.5%	11.5%	975	
Gelcarin GP 812	0.8%	0.3%	25	
Water for gum dispersion		11.5%	975	
Tuna oil	50.0%	17.7%	1500	
Total	100.0%	100.0%	8475	Prepare HWP solution at 60°C, add oligosaccharide and carrageenan solution, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

<b>HWP-oligosaccharide-HMP-MRP (50% tuna oil in powder)</b>				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water		53.3%	4500	
HWP 205	16.7%	5.9%	500	
Raftilose P95	31.7%	11.2%	950	
HMP RS400	1.7%	0.6%	50	
Water for gum dispersion		11.2%	950	
Tuna oil	50.0%	17.8%	1500	
Total	100.0%	100.0%	8450	Prepare HWP solution at 60°C, add oligosaccharide and HMP solution, adjust pH to 7.5, heat to 98°C and hold for 30 minutes, cool down to 60°C, add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

**Example 9: Formulations and Manufacture of Powders with 25% oil loading with blends of sodium caseinate with raw or processed resistant starch (potato starch)**

NaCas-Potato-raw-nonMRP(25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	Prepare 10%TS starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above.
Potato starch	37.5%	5.8%	750	Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	

NaCas-Potato-raw-MRP(25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	Prepare 10%TS starch dispersion at 60°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above.
Potato starch	37.5%	5.8%	750	Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C.
Water for NaCas dispersion		32.7%	4250	Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	

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NaCas-Potato-heat-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	Prepare 10%TS starch dispersion at 60°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Potato starch	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	

NaCas-Potato-heat-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	Prepare 10%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Potato starch	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	

NaCas-Potato-heat-MF-nonMRP(25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Potato starch	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, microfluidise at 800 bar-3 passes. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Potato-heat-MF-MRP(25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Potato starch	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, microfluidise at 800 bar-3 passes. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Potato-extruded-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Potato starch	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS extruded starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

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**Example 10: Formulations and Manufacture of Powders with 25% oil loading with blends of sodium caseinate with raw or pre-processed resistant starch (Hylon VII)**

NaCas-Hylon VII-raw-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hylon VII	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Hylon VII-raw-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hylon VII	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Hylon VII-heat-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hylon VII	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 60°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, and add remaining water to make-up to 10%TS. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Hylon VII-heat-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hylon VII	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

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NaCas-Hylon VII-heat-MF-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hylon VII	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, microfluidise at 800 bar-3 passes. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Hylon VII-heat-MF-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, microfluidise at 800 bar-3 passes. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Hylon VII	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	

NaCas-Hylon VII-extruded-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	Prepare 10%TS extruded starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Hylon VII	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	

**Example 11: Formulations and Manufacture of Powders with 25% oil loading with blends of sodium caseinate with raw or pre-processed resistant starch (Hi-Maize 1043)**

NaCas-Hi-Maize 1043-raw-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	Prepare 10%TS starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Hi-Maize 1043	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	

NaCas-Hi-Maize 1043-raw-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	Prepare 10%TS starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.
Hi-Maize 1043	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	

## NaCas-Hi-Maize 1043-heat-nonMRP (25% tuna oil in powder)

Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hi-Maize 1043	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 60°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

## NaCas-Hi-Maize 1043-heat-MRP (25% tuna oil in powder)

Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hi-Maize 1043	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 60°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

## NaCas-Hi-Maize 1043-heat-MF-nonMRP (25% tuna oil in powder)

Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hi-Maize 1043	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, microfluidise at 800 bar-3 passes. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

## NaCas-Hi-Maize 1043-heat-MF-MRP (25% tuna oil in powder)

Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hi-Maize 1043	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, microfluidise at 800 bar-3 passes. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Hi-Maize 1043-extruded-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hi-Maize 1043	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS extruded starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

**Example 12: Formulations and Manufacture of Powders with 25% oil loading with blends of sodium caseinate with raw or pre-processed resistant starch (Novelose 260)**

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NaCas-Novelose 260-raw-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 260	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Novelose 260-raw-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 260	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Novelose 260-heat-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 260	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Novelose 260-heat-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 260	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Novelose 260-heat-MF nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 260	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, microfluidise at 800 bar-3 passes. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Novelose 260-heat-MF-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 260	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, microfluidise at 800 bar-3 passes. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Novelose 260-extruded-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 260	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS extruded starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

**Example 13: Formulations and Manufacture of Powders with 25% oil loading with blends of sodium caseinate with raw or pre-processed resistant starch (Novelose 330)**

NaCas-Novelose 330-raw-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 330	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	

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NaCas-Novelose 330-raw-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 330	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	

NaCas-Novelose 330-heat-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 330	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	

NaCas-Novelose 330-heat-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 330	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	

NaCas-Novelose 330-heat-MF-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 330	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, microfluidise at 800 bar-3 passes. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Novelose 330-heat-MF-MR (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 330	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, microfluidise at 800 bar-3 passes. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Novelose 330-extruded-MRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Novelose 330	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS extruded starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Heat protein-starch mixture in cans at 98°C-30 minutes, cool down to 60°C. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

5 **Example 14: Formulations and Manufacture of Powders with 25% oil loading with blends of sodium caseinate with raw or high pressure processed or ultrasonicated resistant starch (Hylon VII)**

NaCas-Hylon VII-raw-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hylon VII	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Hylon VII-heat-HPP-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hylon VII	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, HPP at 600 MPa, for 15 minutes. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-Hylon VII-heat-US-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Hylon VII	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, US using 20 KHz unit @ 50 ml per minute, 380 Watts. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

5 **Example 15: Formulations and Manufacture of Powders with 25% oil loading with unheated and heated blends of sodium caseinate with raw starches or pre-processed starch**

NaCas-waxy maize-raw-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Waxy maize	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-waxy maize-heat-MF-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Waxy maize	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, Microfluidise at 800 bar-1 pass. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-maize starch-raw-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Maize starch	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-maize starch-heat-MF-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Maize starch	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, Microfluidise at 800 bar-1 pass. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-wheat starch-raw-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Wheat starch	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	
Total	100.0%	100.0%	13000	Prepare 10%TS starch dispersion at 70°C. Prepare 15%TS NaCas solution at 60°C and mix with starch dispersion above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

NaCas-wheat starch-heat-MF-nonMRP (25% tuna oil in powder)				
Ingredient	% dry basis	% wet basis	Wt of ingredient (g)	Processing steps
Water for starch dispersion		51.9%	6750	
Wheat starch	37.5%	5.8%	750	
Water for NaCas dispersion		32.7%	4250	
Alanate 180	37.5%	5.8%	750	
Tuna oil	25.0%	3.8%	500	Prepare 20%TS starch dispersion at 70°C, process in 73 x 82 mm cans at 121°C-60 minutes, cool down, add remaining water to make-up to 10% TS, Microfluidise at 800 bar-1 pass. Prepare 15%TS NaCas solution at 60°C and mix with processed starch above. Add oil heated to 60°C, homogenise at 350/100 bar, spray dry at 180/80°C Ti/To.

### Characteristics of microcapsules

- The properties of the example 1 formulations are shown in figure 1 of the drawings. Solvent-extractable fat in all powders (25% fat in powder) were less than 3% (of total fat) indicating that the encapsulating efficiency was good. Release of oil in SGF was small (<1.3% of total fat) for all microcapsules. Use of caseinate in combination with the glucose/ dried glucose syrup or oligosaccharide produced microcapsules, which were not degraded by SIF (extractable fat <2% of total fat).
- 5 Encapsulants with caseinate as the protein in the coat were superior to corresponding formulations containing whey protein isolate. Heating of caseinate-carbohydrate mixtures prior to encapsulation did not affect its release properties after sequential exposure to GF and SIF whereas heating of whey protein isolate – carbohydrate mixtures increased oil release.
- 10 The properties of the example 2 formulations are shown in figure 2 of the drawings. Solvent-extractable fat in all powders (50% fat in powder) were less than 3% (of total fat) indicating that the good encapsulating efficiency was maintained when that ratio of the fat to encapsulating material was increased from 1:3 in 25% fat powders to 1:1 in 50% fat powders. The trend in the release properties of the
- 15 microcapsules in Figure 2 with 50% fat powders mirror those observed in Figure 1 for 25% fat powders except that 50% fat powders made with MRP-based encapsulants containing WPI had higher amounts of extractable fat after sequential exposure to SGF and SIF than corresponding formulations for 25% fat powders.

The properties of the example 3 formulations are shown in figure 3 of the drawings.

Formulations (25% fat powders) made with heated protein-starch as encapsulants had low solvent extractable fat (<1% of total fat). All formulations did not release significant amounts of fat in SGF. The type of starch used in combination with

5 caseinate as encapsulant affected release properties. Combination of caseinate with starch (tapioca dextrin K4484) was more effective in reducing release of oil from microcapsules after sequential exposure to SGF and SIF than corresponding WPI-starch formulation.

The properties of the example 4 formulations are shown in figure 4 of the  
10 drawings. Formulations (50% fat powders) made with heated protein-starch as  
encapsulants had higher solvent extractable fat (1.3 –19.9 % of total fat) than  
corresponding formulation for 25% fat powders. All heated caseinate-starch  
formulations did not release significant amounts of fat in SGF and SIF. As was the  
case for 25% fat powders (Figure 3), a combination of caseinate with starch  
15 (tapioca dextrin K4484) was more effective in reducing release of oil from  
microcapsules after sequential exposure to SGF and SIF than corresponding WPI-  
starch formulation (Figure 4). Solvent-extractable fat in powder was not related to  
solvent extractable fat in SGF and SIF fluids.

The properties of the example 5 formulations are shown in figure 5 of the  
20 drawings. Use of gums in combination with protein – glucose/dried glucose syrup or protein –oligosaccharide as encapsulant resulted in powders with low extractable fat in powder (<3.1% of total fat) and SGF (<1.1% of total fat). However, caseinate-based formulations with gums released more fat (figure 5) than similar formulations with without gum (Figure 1) after sequential exposure to  
25 GSF and SIF. As with the other examples (Figures 1-4), caseinate-based formulations were superior to WPI-based formulations for preventing release of oil on exposure to SGF and SIF.

The properties of the example 6 formulations are shown in figure 6 of the drawings. The trends observed for 50% fat powders containing gums in combination with protein-glucose/dried glucose syrup or oligosaccharide (Figure 6) are similar to those observed for compositions with 25% fat powders (Figure 5). However, the amount of release in 50% fat powders (Figure 6) is significantly more than that in 25% fat powders (figure 5) after exposure to SGF and SIF.

The properties of the example 7 formulations are shown in figure 7 of the drawings.

Hydrolysed milk proteins can be used in place of whole proteins for encapsulation of oil. While, combinations of hydrolysed casein with oligosaccharide and gums

5 were less effective for protecting oils from release in SGF+SIF compared to corresponding formulations with the parent protein (Na caseinate), the reverse trend was found with the use of hydrolysed whey protein-base formulations (Compare Figures 5 and 7).

The properties of the example 8 formulations are shown in figure 8 of the drawings. While 10 solvent-extractable fat in powders (50% fat) was low, the hydrolysed casein-based formulation containing carrageenan was not suitable for delivery of cores to the colon as the capsules released a significant amount of their load in SGF but would be suitable if the site for target delivery is the stomach or small intestine. Those containing hydrolysed casein or hydrolysed whey protein with high methoxy pectin 15 were comparatively better at protecting their load than those with carrageenan.

The properties of the example 9 formulations are shown in figure 9 of the drawings. The results above show that 25% fat powders made with unheated and heated combinations of caseinate and raw or pre-processed potato starch had solvent-extractable fat of between 3.7-7.7% of total fat, which was generally higher 20 than those made with combinations of proteins with sugar/dried glucose syrup or oligosaccharides. Exposure to SGF resulted in release of <0.6% of total fat and sequential exposure to SGF and SIF resulted in between 4.5 – 8.4% of total fat being released. Reduction of the resistant starch content of potato starch by pre-processing of the starch did not substantially affect the ability of the 25 microencapsulated formulation to protect the oil from SGF and SIF environments.

The properties of the example 10 formulations are shown in figure 10 of the drawings. The results above show that 25% fat powders made with unheated and heated combinations of caseinate and raw or pre-processed Hylon VII had solvent-extractable fat of between 13-26% of total fat, which was generally higher than 30 those made with combinations of proteins with sugar/dried glucose syrup or oligosaccharides or potato starch indicating that encapsulation efficiencies of formulations with Hylon VII were significantly lower. Use of Hylon VII that had been subjected to microfluidisation or extrusion prior to combination with protein

improved encapsulation efficiency. However, exposure to SGF which results in hydration of the capsule resulted in minimal release of <0.8% of total fat and sequential exposure to SGF and SIF resulted in between 3.1 – 7.1% of total fat being released. Reduction of the resistant starch content of Hylon VII by pre-

- 5 processing of the starch did not substantially affect the ability of the microencapsulated formulation to protect the oil from SGF and SIF environments. The properties of the example 11 formulations are shown in figure 11 of the drawings. The results above show that 25% fat powders made with unheated and heated combinations of caseinate and raw or pre-processed Hi-Maize had solvent-  
10 extractable fat of between 13-26% of total fat, which is similar to those for microcapsules formulated with Hylon VII. Use of Hi-Maize that had been subjected to microfluidisation or extrusion prior to combination with protein improved encapsulation efficiency. However, exposure to SGF which results in hydration of the capsule resulted in minimal release of <0.8% of total fat and sequential  
15 exposure to SGF and SIF resulted in between 3.1 – 7.1% of total fat being released. Reduction of the resistant starch content of Hi-Maize by pre-processing of the starch did not substantially affect the ability of the microencapsulated formulation to protect the oil from SGF and SIF environments.
- The properties of the example 12 formulations are shown in figure 12 of the drawings. The  
20 characteristics of formulations with Novelose 260 were similar to those observed for formulations with Hylon VII or Hi-Maize, which like Novelose 260 are RS2 type starches.

The properties of the example 13 formulations are shown in figure 13 of the drawings. The results above show that 25% fat powders made with unheated and heated

- 25 combinations of caseinate and raw or pre-processed Novelose 330 (an RS3 type starch) had high solvent-extractable fat of between 13-33% of total fat. Of the pre-processing treatments used on starch, only extrusion improved the encapsulation properties of the starch when it was used in combination with protein, which also significantly reduced its RS content. Heating alone or heating used in combination  
30 with microfluidisation did not affect the RS content of Novelose 300. Although solvent extractable fat in powders in the dry state were high, exposure to SGF which results in hydration of the capsule resulted in minimal release of <1% of total

fat and sequential exposure to SGF and SIF resulted in between 3.1 – 8.0 % of total fat being released.

The properties of the example 14 formulations are shown in figure 14 of the drawings. The results above demonstrate that starch pre-processed using

5 emerging food processing technologies (ie high pressure processing or ultrasonication) could be used as encapsulants. Release of oil from microcapsules after exposure to SGF and SIF was lower with use of ultrasonicated starch compared to high pressure processed or raw starch.

The properties of the example 15 formulations are shown in figure 15 of the

10 drawings. The results above demonstrate that use of native non-RS starch and their pre-processed counterparts in combination with protein produced powders with solvent extractable fat of between 5.5-13.6% of total fat. Release of oil from microcapsules after sequential exposure to SGF and SIF was between 12-13.7% (Figure 15), which was higher than that observed when more resistant starches

15 were used in combination with protein for microencapsulation (See Figures 9-14).

From the above those skilled in the art will see that the present invention provides a simple to use yet effective delivery vehicle to the colon as well as preserving sensitive core ingredients during storage and processing. Those skilled in the art

20 will also realise that this invention can be implemented in a number of different embodiments by varying the encapsulant proteins and carbohydrates without departing from the teachings of this invention.

**CLAIMS**

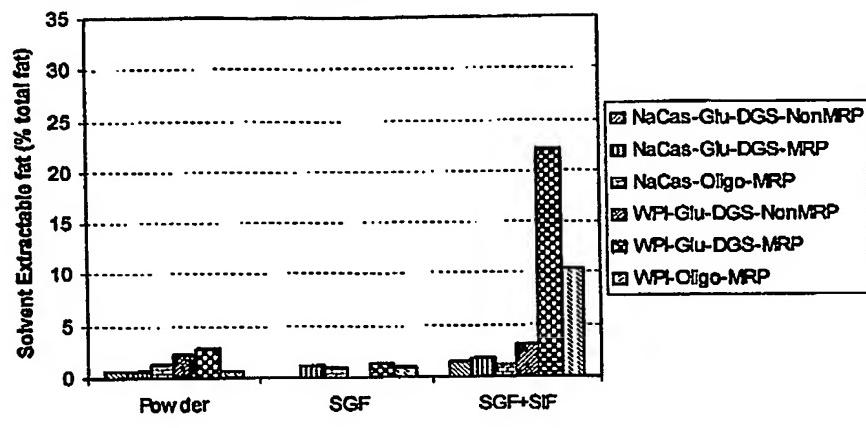
1. A micro encapsulation material for use with storage unstable, therapeutic and nutritional agents which release the therapeutic and nutritional agents in predetermined locations in the gastro intestinal tract in which the  
5 microencapsulation material is formed by combining a food grade treated carbohydrate with a water soluble food grade protein.
2. An encapsulation material as claimed in claim 1 in which the carbohydrate material is treated to make emulsions of the encapsulant material stable and to increase the number of sugar reducing groups in the carbohydrate.  
10
3. An encapsulation material as claimed in claim 1 in which the carbohydrate is selected from those containing reducing sugar groups, oligosaccharides, raw, modified, resistant, acetylated, propionate and butyrate starches.
4. An encapsulation material as claimed in claim 1 in which the protein is selected from milk proteins including casein and whey proteins.  
15
5. An orally administrable nutritional or therapeutic product for delivery of a nutritional or therapeutic agent to the gastrointestinal tract in which the agent includes an oil or an oil soluble or dispersible component which is encapsulated in a material as claimed in claim 1.
6. A method of preparing a nutritional or therapeutic product as defined in  
20 claim 5 which includes the steps
  - a) selecting a nutritional or therapeutic oil, oil soluble or oil dispersible nutritional or therapeutic agent
  - b) dispersing a water soluble film forming protein and a treated carbohydrate in the aqueous phase
  - c) mixing component (a) with component (b) and homogenizing the mixture to obtain an emulsion
  - d) optionally drying the emulsion to obtain a powdered formulation in which the nutritional or therapeutic oil or agent is surrounded by the component (b).  
25

30

**ABSTRACT**

A micro encapsulation material for use with storage unstable, therapeutic and nutritional agents which release the therapeutic and nutritional agents in predetermined locations in the gastro intestinal tract in which the microencapsulation material is formed by combining a food grade treated carbohydrate with a water soluble food grade protein. The therapeutic and nutritional agents form an oil phase which is emulsified with the water dispersed or dissolved encapsulant to encapsulate the therapeutic and nutritional agents. These agents may be oils or oil soluble or oil dispersible. The agents that may be encapsulated include lipids (oils including oxygen sensitive oils, fatty acids, triglycerides) and oil soluble and oil dispersible ingredients (including pharmaceuticals, probiotics, protein therapeutics and bioactives). The protein used may include any film forming water soluble protein or hydrolysed protein and includes milk proteins such as casein and its derivatives or whey proteins. The carbohydrate component may be those containing reducing sugar groups, oligosaccharides and starches (raw, modified, resistant, acetylated, propionated and butyrate starches).

**Figure 1**



**Figure 2**

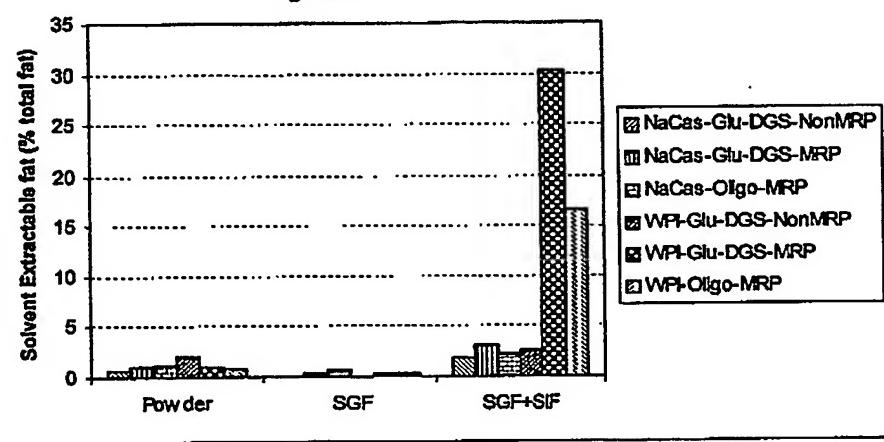


Figure 3

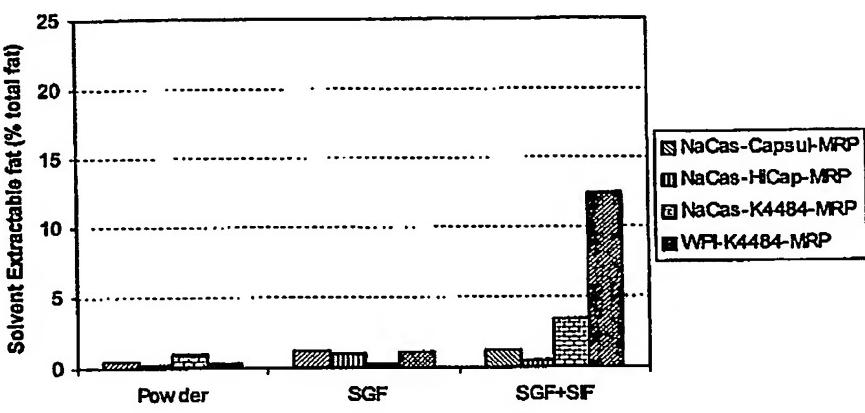


Figure 4

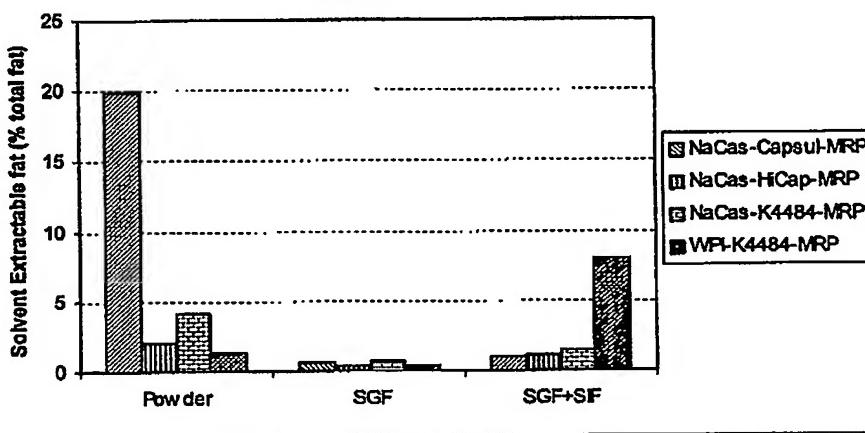


Figure 5

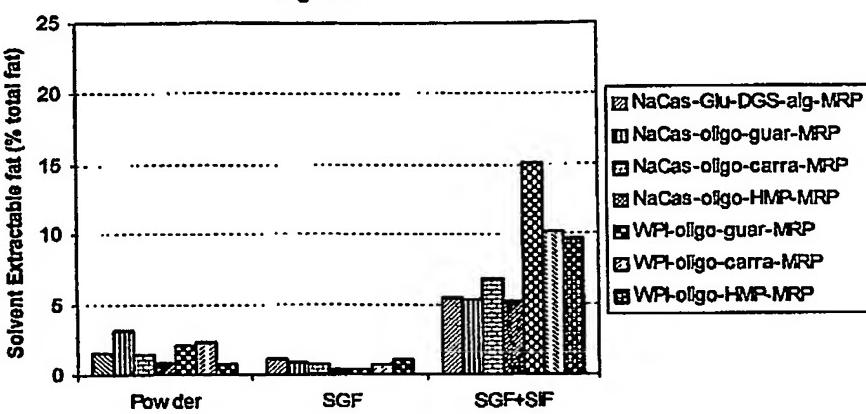


Figure 6

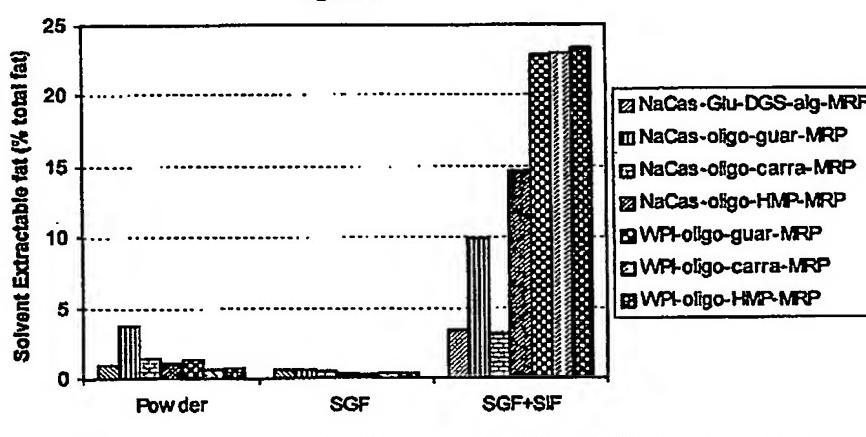


Figure 7

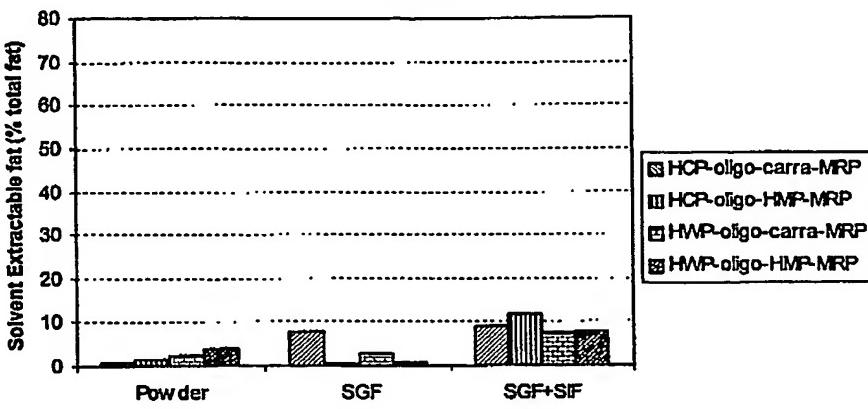
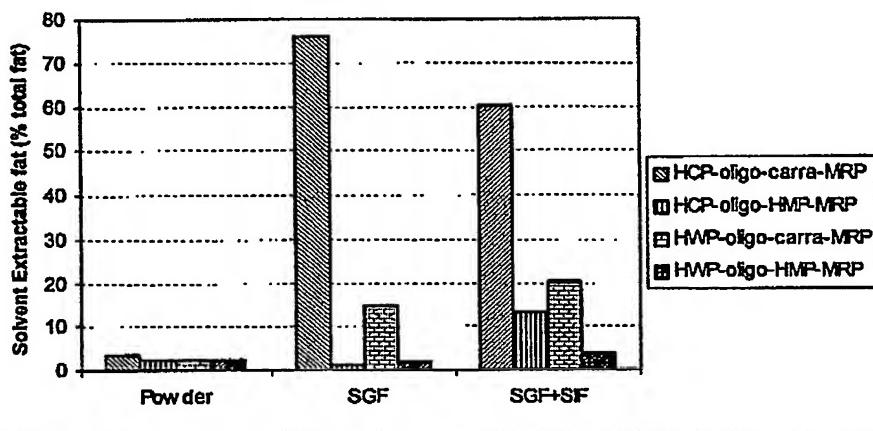
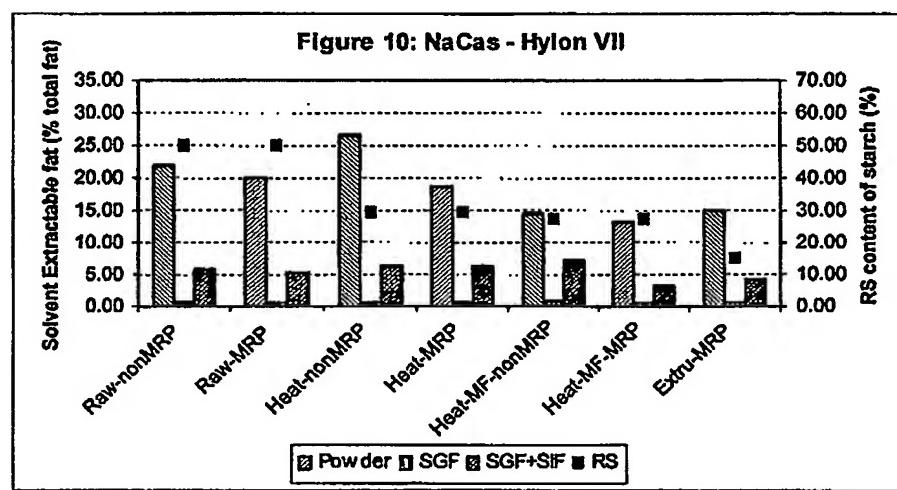
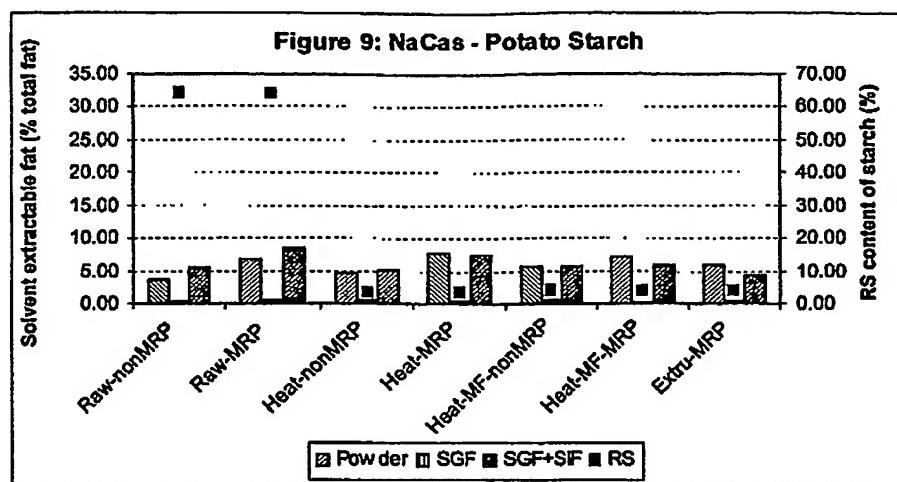
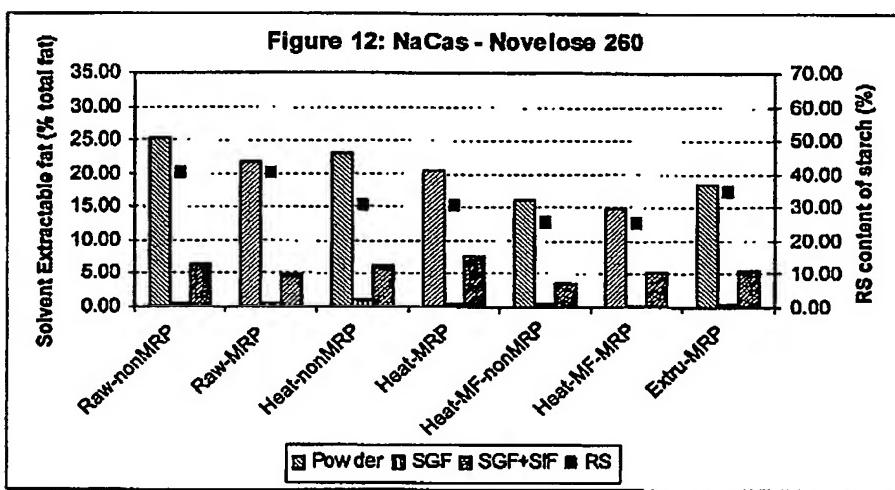
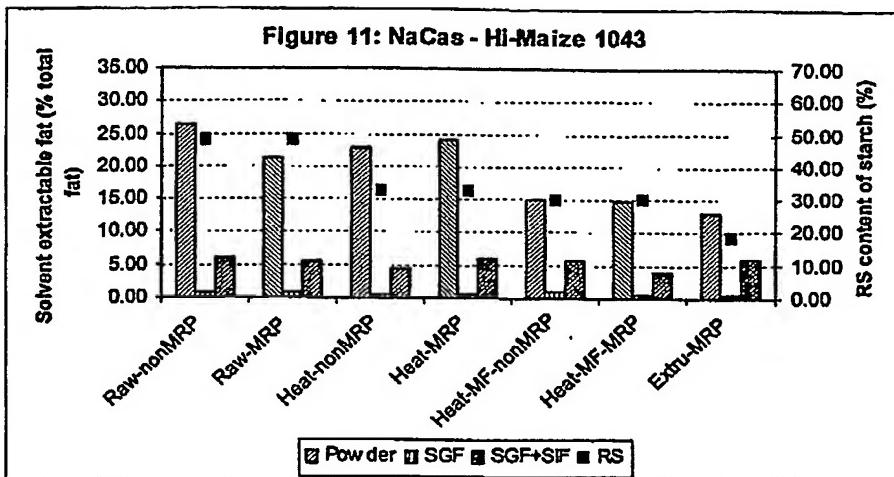
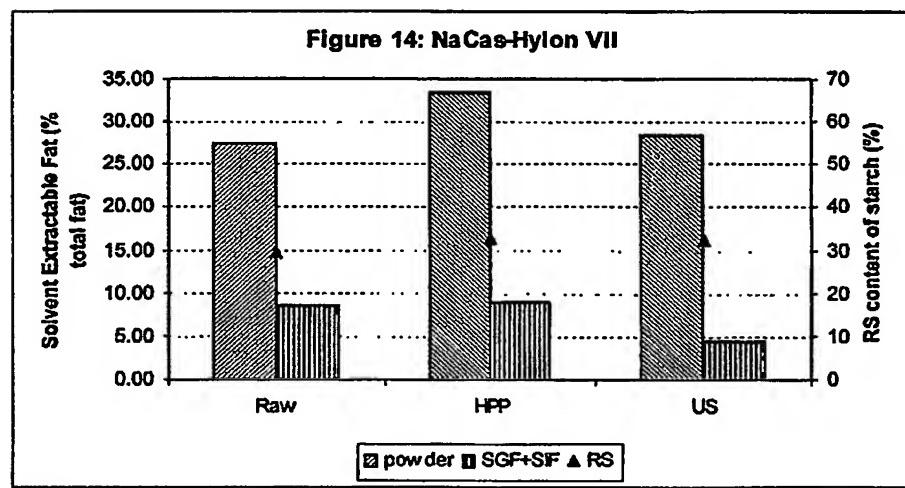
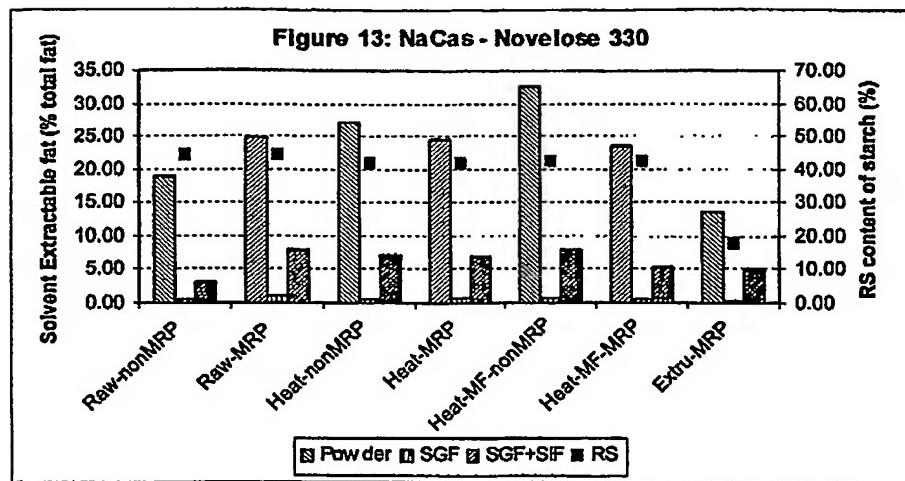


Figure 8

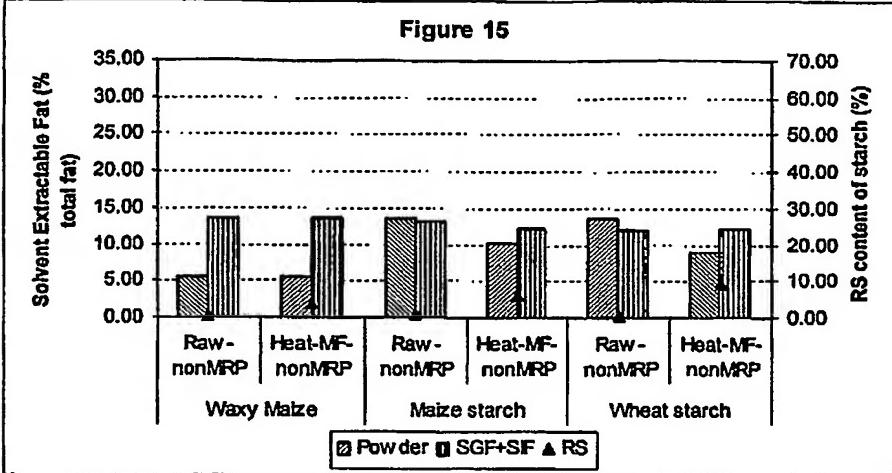








**Figure 15**



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